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# A New Aging Treatment for Improving Cryogenic Toughness of the Main Structural Alloy of the Super Lightweight Tank

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*P.S. Chen and W.P. Stanton*

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November 1996



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*P.S. Chen*

*IIT Research Institute • Chicago, Illinois*

*W.P. Stanton*

*Marshall Space Flight Center • MSFC, Alabama*

National Aeronautics and Space Administration  
Marshall Space Flight Center • MSFC, Alabama 35812

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## CHEMICAL SYMBOLS

Al aluminum

Li lithium



## TECHNICAL MEMORANDUM

# A NEW AGING TREATMENT FOR IMPROVING CRYOGENIC TOUGHNESS OF THE MAIN STRUCTURAL ALLOY OF THE SUPER LIGHTWEIGHT TANK

## INTRODUCTION

NASA has selected Al-Li alloy 2195 to be the main structural alloy of the super lightweight tank (SLWT) for the space shuttle. This alloy has significantly higher strength than conventional 2XXX alloys (such as 2219) at both ambient and cryogenic temperatures. If properly processed and heat treated, this alloy can display higher fracture toughness at cryogenic temperature than at ambient temperature. However, the properties of production materials have shown greater variation than those of other established alloys, as is the case with any new alloy that is being transitioned to a demanding application. To ensure proper quality control, NASA has imposed lot acceptance testing on alloy 2195 plate before it can be used in the SLWT program. During lot acceptance testing, strength and toughness are measured at ambient and cryogenic temperatures in relevant positions and orientations.

Currently, some commercial 2195 plate for the SLWT program is being rejected, mostly due to low cryogenic fracture toughness (CFT) or fracture toughness ratio (FTR) at ambient and cryogenic temperatures. As a result, an in-house scientific study was initiated to understand and correlate FTR with alloy composition and microstructure. This study clearly indicated that the size and density of  $T_1$  precipitate are key factors affecting CFT. The most interesting findings included:

1. Alloy 2195 can display high cryogenic toughness and an FTR greater than 1.05 when subgrain boundaries contain no more  $T_1$  than the matrix.
2. CFT and FTR dropped sharply when  $T_1$  precipitated preferentially at subgrain boundaries, decreasing as  $T_1$  coverage increased (see figure 1).

In late 1994, another in-house effort was initiated to develop a new thermal processing technique that can control the size and location of  $T_1$  precipitate, in order to achieve higher CFT and FTR. Ultimately, it is hoped that this new technique can be exploited to reduce the rejection rate of unqualified materials, making the SLWT program more cost-effective. To date, a new aging schedule has been developed that can prevent  $T_1$  from preferentially precipitating at subgrain boundaries.<sup>1,2</sup> When tested, the aging treatment proved to be very effective at enhancing CFT and FTR in "good," "marginal," and "bad" lots of alloy 2195. This report details the effects of the new aging technique on these parameters.

## TECHNICAL APPROACH

At present, alloy 2195 plates are being aged for the SLWT program using conventional isothermal aging at either 290 or 300 °F, for times varying from 25 to 35 h. This conventional aging process ages up the materials in a reasonable period of time, but it tends to promote  $T_1$  to precipitate preferentially at subgrain boundaries, leading to unacceptably low CFT and FTR. Lowering the aging temperature to 280 °F or lower is one way to avoid excessive  $T_1$  precipitation at subgrain boundaries. However, this tactic also resulted in a significant drop in yield strength, which then did not meet the minimum requirement of 73 ksi. In addition, low-temperature aging is associated with sluggish aging kinetics, which are not desirable for industrial mass production.

Therefore, a new aging treatment was designed to improve CFT and FTR without sacrificing yield and tensile strength. It requires multiple steps, and the heating rate is precisely controlled to meet target properties. The new aging treatment consists of the following steps (see figure 2):

1. Hold at 260 °F for 10 h
2. Heat continuously from 260 to 275 °F at a rate of 1 °F/h
3. Hold at 275 °F for 10 h
4. Heat continuously from 275 to 290 °F at a rate of 1 °F/h
5. Hold at 290 °F for 20 h to obtain a near peak-aged condition.

The resulting fracture toughness is considerably higher than that of isothermally aged alloy at similar levels of yield strength, thus producing excellent strength/toughness combinations.

Early in this program, design of experiments (DOE) ingot No. 10 was used for aging process development and schedule optimization. The new aging treatment was found to be very effective, improving CFT by approximately 15 to 20 percent. Table 1 and figure 3 show a head-to-head comparison of conventional aging with the new treatment (using DOE ingot No. 10), while table 2 details the heat treatment schedule used for DOE Ingot No. 10.

In order to further evaluate the repeatability and effectiveness of this new aging treatment, investigators then selected and tested three more lots of alloy 2195, in the form of 1.75-in-thick gauge plates with FTR values ranging from 0.85 to 1.07. The new aging treatment again demonstrated its effectiveness in improving CFT and FTR.

## RESULTS

### Fracture Toughness and Strength

The SLWT program requires a minimum strength of 73 ksi. Tensile data indicated that the new aging treatment can achieve the same yield strength levels as those produced by conventional aging (table 2). The most encouraging improvements were seen in CFT and FTR, for which the minimum requirements are 30 ksi/in and 1.0, respectively. As shown in Table 1 and figure 3, the new aging treatment significantly improved both CFT and FTR in DOE ingot No. 10. The same trend was observed on these three lots of production material. After conventional aging, lot (F) possessed high FTR. The new aging treatment raised its absolute CFT values and led to a slight increase in FTR. Lots (A) and (B) exhibited FTR values less than 1. The new aging treatment significantly improved their CFT and FTR values (table 3). Figures 4 and 5, respectively, show a detailed comparison of CFT and FTR for all three lots.

### Microstructure

Transmission electron microscopy (TEM) indicated that the effectiveness of this new aging treatment lies in its ability to control the location and size of strengthening precipitate  $T_1$ , as observed in DOE ingot No. 10. Figure 6 compares the subgrain boundary microstructure for material from lot (A), which was aged using the conventional and new aging treatments. The new aging treatment greatly reduced the degree of  $T_1$  precipitation at subgrain boundaries.

## Benefits to SLWT Program

In addition to improving CFT and FTR, the new aging treatment also reduced statistical spread of fracture toughness values and FTR, greatly reducing the disparity between good and bad lots (figs. 5 and 6). If implemented, the new aging process can improve SLWT reliability, decrease the rejection rate for unqualified materials, and ultimately reduce NASA's costs for this high-value material.

The new aging treatment is not recommended for "good" lots, due to the fact that the entire aging duration (approximately 60 h) is much longer than that of conventional aging (approximately 30 h), which is normally quite adequate to obtain acceptable properties. Instead, the new aging treatment should be exploited to process "bad lots" in order to avoid excessive material rejection due to low toughness and FTR. NASA will be most benefited by using both aging treatments to prevent potential material shortages and launch schedule slips.

## SUMMARY

1. This new aging treatment achieves high strength by promoting  $T_1$  nucleation in the matrix, so that the total number density of  $T_1$  is higher than that seen in conventionally aged materials. The new treatment reduces the length of time that the materials are exposed to high temperatures, constraining  $T_1$  nucleation and growth at subgrain boundaries and permitting the material to achieve much improved cryogenic fracture toughness.
2. The new aging treatment was designed to process alloy 2195, in order to improve fracture toughness, reduce statistical spread of strength and fracture toughness, decrease rejection rates, and reduce material cost. Its only disadvantage is a longer aging duration (approximately 60 h) compared to conventional isothermal aging (approximately 30 h). However, if the rejection rate can be minimized for this high-value material, the cost advantages will clearly outweigh any disadvantages associated with longer aging duration.

## REFERENCES

1. Chen, P.S., Kuruvilla, A.K., Malone, T.W., and Stanton, W.P.: "Improving Cryogenic Toughness of Alloy 2195 by Optimizing Aging." Submitted to J. Mater. Sci. Eng. for publication, 1995.
2. Chen, P.S., Kuruvilla, A.K., Malone, T.W., and Stanton, W.P.: "A Multi-Step Heating Rate-Controlled Aging Treatment for Improving Cryogenic Toughness of Aluminum-Lithium Alloys," 1994, patent application filed, 1995.

Table 1. New aging treatment improves CFT and FTR for DOE ingot No. 10.

Heat Treatment	Aging Treatment	YS (ksi)	UTS (ksi)	%El	K at a/2 (Ambient)	K at a/2 (Cryo)	FTR (Cryo/Ambient) *
No. 1	Conventional	75.4	78.9	7.4	31.17	28.96	0.93
No. 2	Conventional	77.0	79.8	8.1	27.83	27.62	0.99
No. 3	Conventional	73.1	76.7	12.2	32.09	33.10	1.03
No. 4	Conventional	75.5	78.1	7.9	30.74	30.19	0.98
No. 5	New	77.0	80.2	7.9	30.40	31.70	1.04
No. 6	New	73.9	77.1	10.4	31.00	33.90	1.09

\* Ratio of cryogenic toughness to ambient temperature toughness

Table 2. Aging treatment used for alloy 2195 (DOE ingot No. 10).

Specimen	Stretch	Aging Treatment
No. 1	6%	SHT + 300 °F/18 h
No. 2	6%	SHT + 290 °F/30 h
No. 3	6%	SHT + 300 °F/14 h
No. 4	6%	SHT + 290 °F/26 h
No. 5	3%	SHT + 260 °F/10 h + CR to 275 °F (1 °F/h) + 275 °F/10 h + CR to 290 °F (1 °F/h) + 290 °F/25 h
No. 6	3%	SHT + 260 °F/10 h + CR to 275 °F (1 °F/h) + 275 °F/10 h + CR to 290 °F (1 °F/h) + 290 °F/15 h

\* SHT: solutioning heat treatment

\* CR: continuous ramping at 1 °F/h

Table 3. Aging treatment and mechanical properties of alloy 2195.

Lot No. (3% Stretch)	Aging	YS (ksi)	UTS (ksi)	%El	K at a/2 (LN2)	K at a/2 (Ambient)	FTR (Cryo/Ambient)
(A)	Modified	77.8	84.6	8.1	34.71	31.95	1.090
	Conventional	76.5	84.4	8.0	29.59	30.64	0.966
(B)	Modified	77.8	85.1	9.4	32.10	31.80	1.010
	Conventional	74.0	83.1	7.0	25.40	30.04	0.846
(F)	Modified	76.1	83.0	9.1	37.13	34.50	1.080
	Conventional	76.1	83.4	8.0	34.91	32.90	1.060

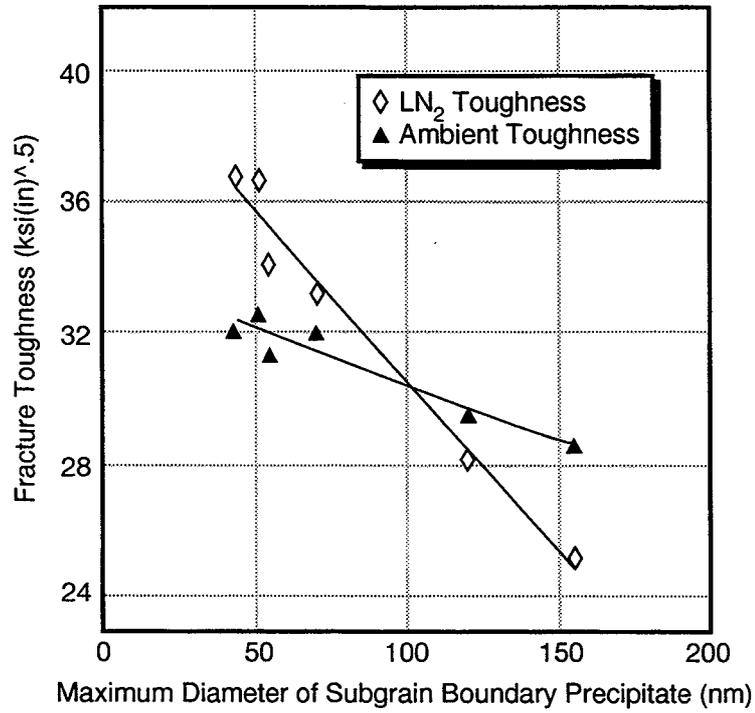


Figure 1. Fracture toughness versus maximum size of T1 at subgrain boundaries, with fracture toughness decreasing as T1 size increases.

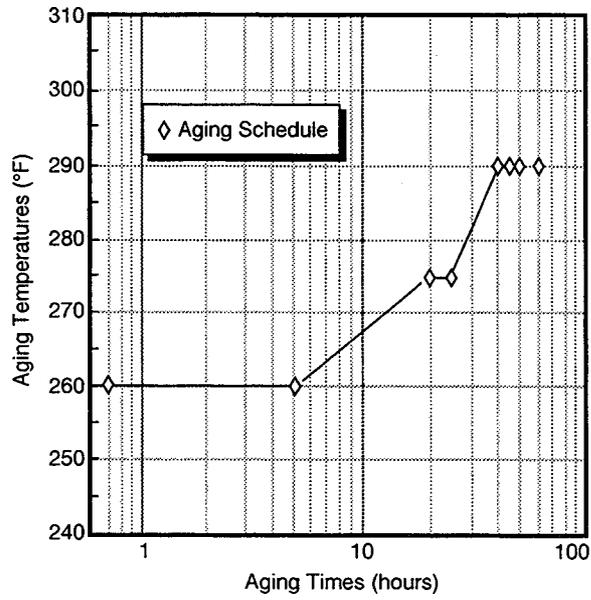


Figure 2. Schedule for multistep and rate-controlled aging treatment.

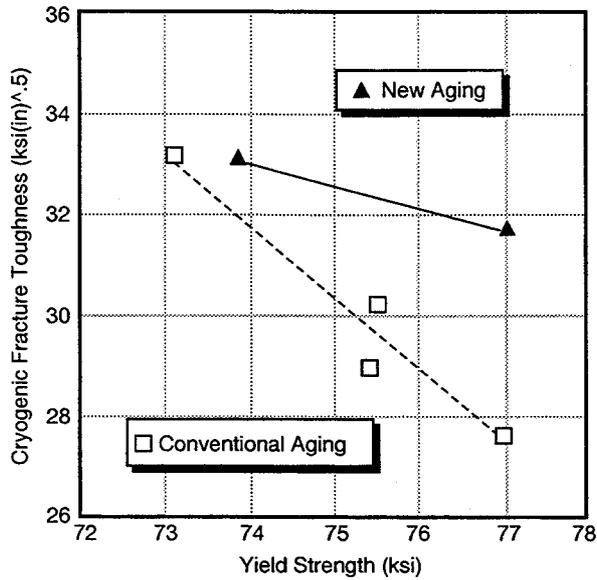


Figure 3. Fracture toughness as a function of yield strength using conventional and new aging treatments. Note that significant improvement on fracture toughness (especially at cryogenic temperatures) was made using the new aging treatment.

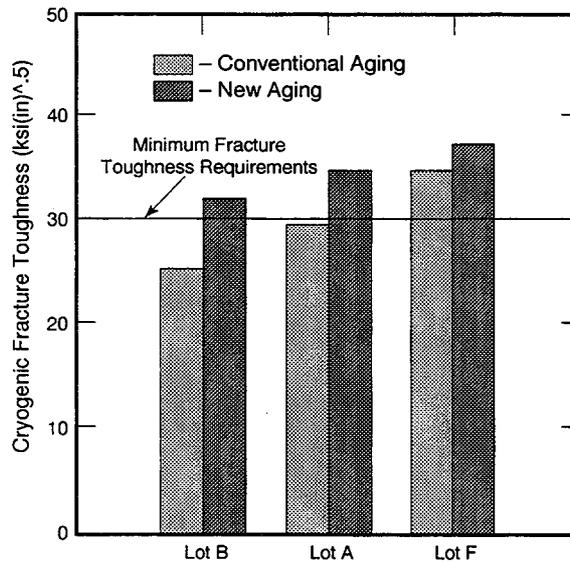


Figure 4. Cryogenic fracture toughness data obtained using conventional and new aging treatment. As shown, the two initially rejectable lots (A) and (B) become acceptable by meeting the minimum fracture toughness requirements.

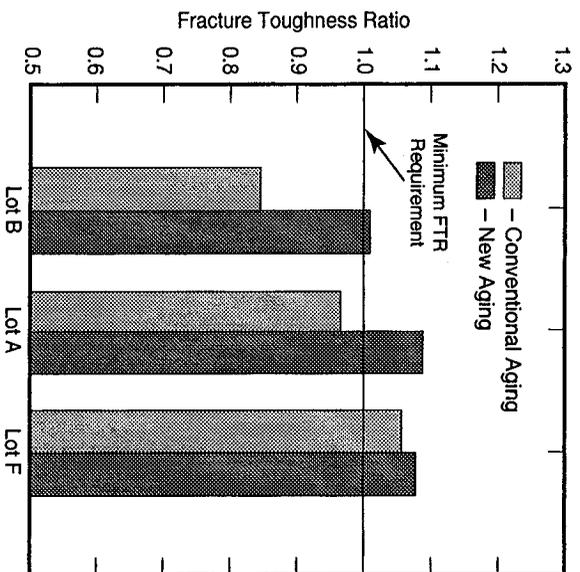


Figure 5. Fracture toughness ratio data obtained using conventional and new aging treatment. As shown, the two initially rejectable lots (A) and (B) become acceptable by meeting the minimum FTR requirements.

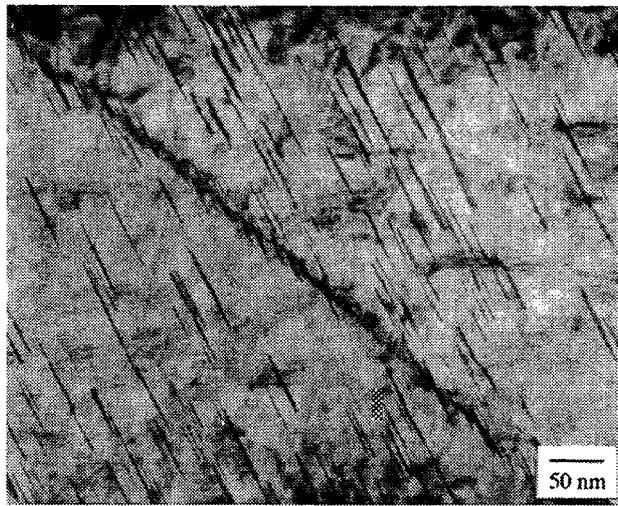


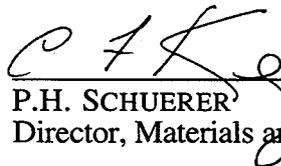
Figure 6. TEM micrographs showing subgrain boundary microstructure of lot (A) aged by conventional isothermal aging (top) and the new aging treatment (bottom).

## APPROVAL

### A NEW AGING TREATMENT FOR IMPROVING CRYOGENIC TOUGHNESS OF THE MAIN STRUCTURAL ALLOY OF THE SUPER LIGHTWEIGHT TANK

By P.S. Chen and W.P. Stanton

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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P.H. SCHUERER  
Director, Materials and Processes Laboratory

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13. ABSTRACT (Maximum 200 words) Marshall Space Flight Center (MSFC) has developed a new technique that can enhance cryogenic fracture toughness and reduce the statistical spread of toughness values in alloy 2195. This aging treatment can control the location and size of strengthening precipitate T1, making improvements possible in cryogenic fracture toughness (CFT) and fracture toughness ratio (FTR). At the start of this program, design of experiments (DOE) ingot No. 10 was used as a baseline for aging process development and optimization. The new aging treatment was found to be very effective, improving CFT by approximately 15 to 20 percent for DOE ingot No. 10. To further evaluate the repeatability and effectiveness of this new treatment, the investigators selected and tested three more lots of alloy 2195, using 1.75-in-thick gauge plates with FTR values ranging from 0.85 to 1.07. The new aging treatment effectively enhanced CFT and FTR values for all three lots. In one instance, the material was considered rejectable because it did not meet the minimum FTR value (1.0) of the super lightweight tank (SLWT). The new aging treatment improved its FTR from 0.85 to 1.01, making this material acceptable for use in the SLWT.				
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